

Incident Investigation

1) Incident Number:

2) Title: 17-243, 5354 Suspension Loss

3) Classification: Major Incident

4) Estimated Dollar Loss: *Total: \$*

Raw Material costs: \$

Reactor Clean Out: \$

Lost production \$

(hrs/13 hr per batch*58,775 lbs/ batch * .06 per lb)

5) Nature of Damage: Lost suspension and dumped to the containment pit

6) Date and Time of Incident: 05/21/12, 03:54 trip; bad sample at approximately 6:09 am.

7) Date of Investigation: 05/21/12 – Operators on shift, technical staff

8) Location: EPS Building 4, Reactor 17

9) Description of Incident:

On 05/21/11, batch 17-243 in Reactor 17 was charged at 02:43. This was the third batch of 5354 in this reactor. This was the second batch with Arkema BPO. The first batch with Arkema was killed approximately 2 hours and 36 minutes into react. All lot numbers for this batch's charges were the same as the previous batch. TCP was increased by one pound for this batch.

The batch timer began at 03:54. A profile alarm was received at 5:53 and 6:03. The first sample was pulled by the day crew at 5:54 right after shift change and was 800 microns. The second sample was pulled at 6:08. This sample was approximately 1200 microns. The batch was killed at this time. A profile alarm was received at 6:13, 6:14, 6:23 and 6:24. The third sample was 1300 microns taken at 6:24 and the batch was killed again. Emergency cooling was applied at 6:31. The operators sampled the reactor and saw "snot".

One shot of PVA was attempted at 6:34. The batch temperature was 200.7 °F. An EPVA valve error position hi alarm was received 30 seconds after the request to open the EPVA valve. At 6:37 a second attempt to open the EPVA valve was made. An EPVA valve error position hi alarm was received again after the request to open the EPVA valve. The PVA transfer was aborted and restarted at 6:38. Again the EPVA valve error was received. A final attempt at adding PVA was made at 6:46 with the same error being received. According to the level drops in the PVA tank initiated at these times 1130, 1188, 1057 and 1119 pounds of PVA were added respectively. The operators resampled the batch. There was still no suspension. The jacket temperature was dropping and the reactor temperature was rising. The operators opened the emergency cooling bypass to the jacket.

The operators sampled again and suspension was regained. The amps in the reactor were not going up and the temperature did not start rising until 7:22. 2000 lbs of cold water were added to the reactor at 6:53, 7:02 and 7:18 to keep the reactor temp below 200 °F. The R17 batch was

shutdown at 7:50. Agitator current hi alarm received at 8:07. The agitator amps were 190.2. The agitator RPMs were lowered from 54 to 50 at 8:08. Additional agitator current hi alarms received until agitator amps peaked at 250.4 and dropped to 184 at 8:08. Hot drop was initiated at 8:11 to make room for the second TCP charge. The reactor level was 8 inches and remained stuck at this level for the remainder of the incident. The reactor contents were dropped for 10 minutes until 8:21. Agitator current hi alarm received at 8:17. Agitator amps were 193. Last goodies addition was initiated at 8:18. The bottom cat tank valve would not open until 8:24. The jacket supply set point was lowered to 50 °F at 8:37. Around this time, a thumping noise and excessive vibration started in R17, thus the batch was dumped to the pit at 8:39 at 193 °F. The batch was 4 hours and 45 minutes along.

R17 pressure control was set to 5 psig at 8:39. The agitator RPMs were set to 13 at 8:42. Agitator speed control deviation high received at 8:42. High agitator amp alarm received at 8:44 (amps were 189). Agitator amps increased from 189 at 8:44:03 to 410.8 at 8:47:45. Agitator speed control deviation low received at 8:46. 2000 pounds of water added at 8:46. R17 temperature differential alarm received at 8:47. The top temperature was 187 °F and the bottom temp was 190 °F. Agitator current high alarm received at 8:47:58.

After the event, it was confirmed that the two surfactant additions were properly made. The A-81 was still on the table when suspension was lost, indicating that it had not been charged early. Batch conditions including level after each charge, water conductivity, and additives that are weighed on the scale were verified to be correct. All raw materials used in the batch were successfully used on other batches of this product type previously. Subsequent batches had a BPO lot change in R16 and R18 with all other raw materials the same. R18 batches were without issue; however, all subsequent R16 batches were killed.

10) Contributing Factors:

- i. Shift change occurred when sampling of this batch started.
- ii. This reactor jacket was running slightly cooler than the profile from shortly after trip. This indicates there was some issue with the suspension of the batch from the beginning.

11) Root Causes:

Unknown at this time. This indicates an issue with PK suspension stability.

12) Recommendations and Corrective Actions:

13) Investigation Team:

Investigation Facilitator (for taproot)
Steve Bordwine, Process Engineer
Mike Wilke, Process Operator
Steve Emmett, Operations Manager
Doug Thurman, Production Facilitator
Jeff Peterson, Process Operator
Roy Leckonby, TD Team Leader
Ed Zielinski Process Operator